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14. ABSTRACT  Cross sections and branching ratios are presented for the dissociative recombination of the CF <sub>2</sub> <sup>+</sup> and CF <sub>3</sub> <sup>+</sup> ions with electrons. It is found that the channel producing CF + F is dominant for the reaction with CF <sub>2</sub> <sup>+</sup> and the production of CF <sub>2</sub> + F is dominant for the reaction with CF <sub>3</sub> <sup>+</sup> . The cross sections for these two ions are very similar.					
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## The dissociative recombination of fluorocarbon ions III: $\text{CF}_2^+$ and $\text{CF}_3^+$

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### Abstract

Cross sections and branching ratios are presented for the dissociative recombination of the  $\text{CF}_2^+$  and  $\text{CF}_3^+$  ions with electrons. It is found that the channel producing  $\text{CF} + \text{F}$  is dominant for the reaction with  $\text{CF}_2^+$  and the production of  $\text{CF}_2 + \text{F}$  is dominant for the reaction with  $\text{CF}_3^+$ . The cross sections for these two ions are very similar.

### 1. Introduction

Knowledge of the plasma chemistry of  $\text{CF}_4$  is a subject of great importance in the semiconductor industry as it is widely used as an etching gas. Compilations of cross sections and rate coefficients for the many and varied reactions that occur in a  $\text{CF}_4$  plasma have been given by Christophorou and co-workers [1, 2], but until recently there was no information concerning certain reactions, in particular that of the dissociative recombination of ions formed in the  $\text{CF}_4$  plasma. We have recently, however, published cross sections for the ion  $\text{CF}^+$  [3] measured using the ASTRID and CRYRING storage rings. A flowing afterglow Langmuir probe-mass spectrometer (FALP-MS) measurement of the rate coefficient of the recombination of  $\text{CF}_3^+$  at 300 K has also been published [4]. In this paper, we have continued this study with

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storage ring investigations of the cross sections and branching ratios for the dissociative recombination of  $\text{CF}_2^+$  and  $\text{CF}_3^+$ .

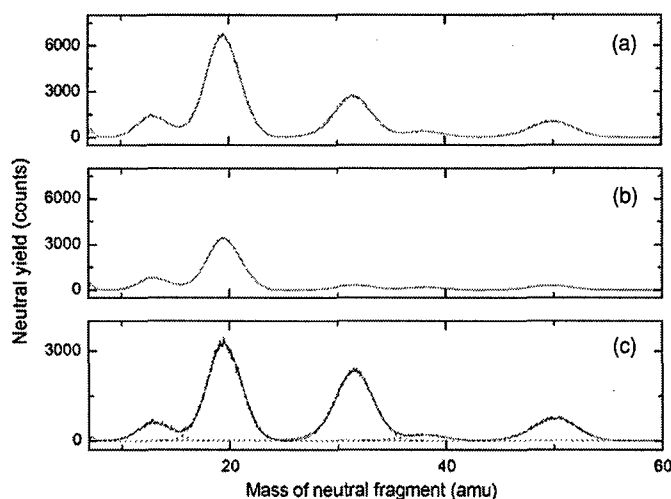
For these two ions, the following dissociation channels are energetically allowed at zero relative energy, assuming that the parents and products are in their ground states. All energies are calculated using data from the NIST Webbook [5], except for the value of the  $\text{CF}_3^+$  heat of formation, which is deduced from the appearance energy of  $\text{CF}_3^+$  from  $\text{CF}_4$  [6]:



## 2. Experimental method

The experiments on  $\text{CF}_2^+$  were performed at the ion storage ring CRYRING, Manne Siegbahn Laboratory, Stockholm University, Sweden and the  $\text{CF}_3^+$  measurements were made at the ASTRID ion storage ring, University of Aarhus, Denmark. The two storage rings have similar features, and the experimental procedures differ only slightly between them. A general description of the apparatuses and procedures can be found in previous papers [7, 8]. In both experiments, the ions were produced from  $\text{CF}_4$  parent gas in a Nielsen-type ion source [9]; the  $\text{CF}_2^+$  was created without a filament at high pressure (about 0.1 Torr) and with an electrical discharge of 1 kV, whereas the  $\text{CF}_3^+$  was produced in the more standard way with a filament and low pressure (about 1 mTorr). After extraction from the ion source at 40 kV (CRYRING) and 150 kV (ASTRID), the ions were injected into the storage rings and further accelerated by RF units to the maximum energies of 1.92 MeV and 1.95 MeV respectively. At that energy the ions were stored for a few seconds, the lifetimes of the beams being 1.4 s and 2 s, respectively, which allowed infrared relaxation to 300 K vibrational distributions. During this storage time, the ions were merged with a monoenergetic electron beam formed in the electron cooler, the parallel overlapping region between the ion and electron beams being 85 cm at CRYRING and 100 cm at ASTRID. In these experiments, the velocity of the electrons can be adjusted to achieve velocity matching between the electrons and the ions while the ion velocities are held constant. During this condition, the velocity spread of the ions is reduced due to the Coulomb interaction with the electrons, a process known as electron cooling [10]. The acceleration region of each electron cooler, a conventional magnet at ASTRID and a superconducting magnet at CRYRING, leads to adiabatic expansions of the electron beams which serve to reduce their transverse energy spreads [11]. The longitudinal energy spreads are reduced due to kinematic compression [10]. It is estimated that the transverse and longitudinal energy spreads are 2 meV and 0.1 meV respectively at CRYRING and 25 meV and 0.5 meV at ASTRID.

The neutral products from the electron-ion collisions and from ion-background gas reactions are unaffected by the dipole magnets in the storage ring and continue in a straight line to energy sensitive semiconductor detectors situated downstream of the interaction regions. These detectors have unitary detection efficiency [12], and the transverse separation between the DR fragments is small enough to ensure that all particles strike the detector. At CRYRING, the products from collisions with the rest gas are monitored using a micro channel plate (MCP) in a separate straight section of the ring. This signal is proportional to the total ion current in the ring and is used for calibration purposes as discussed later.



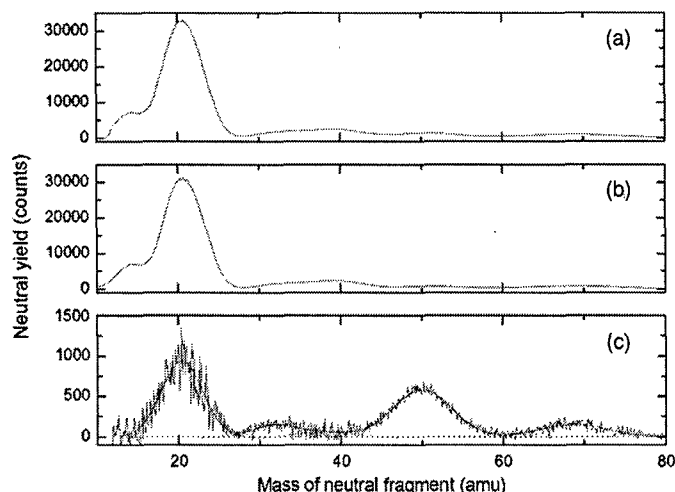
**Figure 1.** The neutral yield from the DR of  $\text{CF}_2^+$  at 0 eV collision energy, recorded with a transmission grid in front of the detector. (a) Fragments from DR and background processes. (b) Background spectrum measured at 1 eV relative energy. (c) Spectrum showing only DR. Dashed line represents Gaussian fitting to each peak.

### 3. Product branching fractions

In order to determine the DR product distribution of  $\text{CF}_2^+$  and  $\text{CF}_3^+$  ions, the neutral fragments created in the electron cooler sections were detected with the semiconductor detectors. After linear amplification, the signals were recorded with multi channel analysers, giving the pulse height spectra of the signals. The products from a dissociation event have a total energy equal to the energy of the ion beam, and the velocity difference between the fragments is such that the difference in the times of arrival is shorter than the response of the detector. Therefore, the molecules from each dissociation event will only give a signal at the full ion energy. In order to separate the fragments, grids with transmissions  $T = 0.297$  at CRYRING and  $T = 0.235$  at ASTRID were inserted in front of the detector. The DR product pulse height spectra at 0 eV collision energy of  $\text{CF}_2^+$  and  $\text{CF}_3^+$ , recorded with these transmission grids in front of the detectors, are shown in figures 1(a) and 2(a), respectively. In order to subtract the background contribution, coming mainly from rest gas collisions, spectra were recorded with the electrons either tuned to a relative energy of 1 eV (CRYRING), where the signal can be considered to come only from background processes [13], or with the electrons turned off (ASTRID). These spectra are shown in figures 1(b) and 2(b). At CRYRING this was done in two separate measurements which were normalized to each other using the MCP detector described previously, whereas at ASTRID the electrons were turned on and off by fast switching. The signal due to DR is shown in figures 1(c) and 2(c), respectively. To extract the product distributions from the recorded spectra, the peaks were fitted and an equation system using the transmission of the grid and the peak areas was applied [14].

### 4. Cross section

To obtain the DR cross section for different centre-of-mass energies, the collision energy between the ions and the electrons was changed by varying the electron energy. The procedure



**Figure 2.** The neutral yield from the DR of  $\text{CF}_3^+$  at 0 eV collision energy, recorded with a transmission grid in front of the detector. (a) Fragments from DR and background processes. (b) Background spectrum measured with the electrons turned off. (c) Spectrum showing only DR. Dashed line represents Gaussian fitting to each peak.

for doing so differed slightly between CRYRING and ASTRID. In the study of  $\text{CF}_2^+$  at CRYRING, the electron energy was linearly ramped during 1 s from an energy corresponding to 1 eV collision energy (electrons moving faster than ions) through zero (velocity matched conditions) to 1 eV collision energy again (electrons slower than ions). At 1 eV collision energy, the DR cross section is very low, and the signal can be considered to come only from background processes [13]. This can therefore be used to find the exponential decay of the background and subtract it from the signal.

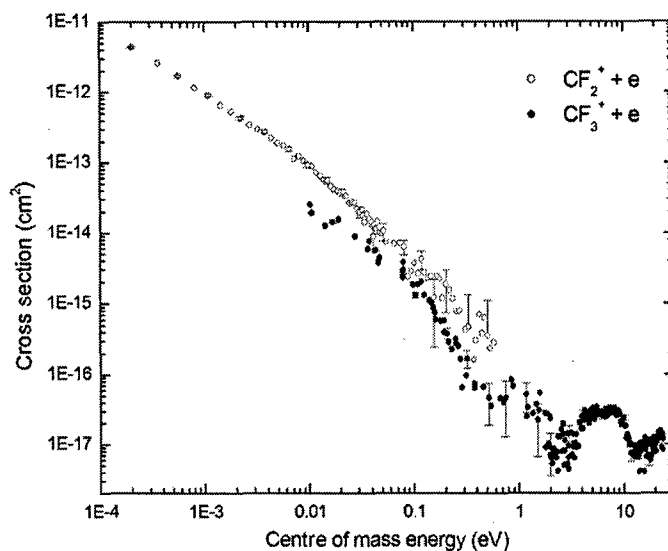
In the measurement of  $\text{CF}_3^+$  at ASTRID, relative energies up to 24 eV were studied in order to search for higher lying resonances. In these measurements a jump method was applied, where the electron beam was chopped on and off during the scan of the electron energy, thereby accumulating background and signal including background simultaneously.

At CRYRING the absolute ion current was measured using a Bergoz beam charge monitor, with continuous averaging and an integrating current transformer [15]. An absolute measurement of the ion current was made with a higher current (91 nA) than was used for the DR measurements (6 nA). Simultaneously with this measurement, the products from collisions with background gas were monitored using the MCP detector described previously. During the DR measurements, the same procedure was followed, and the absolute current could be achieved by normalization of the spectra from the MCP monitoring the background collisions.

At ASTRID, the current was measured using pickup electrodes in the ring, which was calibrated for a known ion current in a separate run [16]. The method used there, however, is less accurate and only gives an order of magnitude estimation of the cross section.

## 5. Results and discussion

The main product channel for DR of both  $\text{CF}_2^+$  and  $\text{CF}_3^+$  was found to be the dissociation into a single F atom and CF or  $\text{CF}_2$ , respectively. The branching fraction for the channel  $\text{CF} + \text{F}$  was determined to be  $71 \pm 4\%$  for  $\text{CF}_2^+$  and  $80 \pm 10\%$  for the channel  $\text{CF}_2 + \text{F}$  for  $\text{CF}_3^+$ .



**Figure 3.** The experimental DR cross section for  $\text{CF}_2^+$  (○) and  $\text{CF}_3^+$  (●). Error bars represent statistical errors of the measurements. The absolute value of the  $\text{CF}_3^+$  data is uncertain (see the text). In the range 0.01–0.1 eV we have assumed that it is equal to that for  $\text{CF}_2^+$ , but we have left the shift in the figure to distinguish the two data sets and to emphasize their similarity.

The channel leading to  $\text{C} + 2\text{F}$  accounts for  $29 \pm 4\%$  for  $\text{CF}_2^+$ , while the corresponding channel ( $\text{CF} + 2\text{F}$ ) for  $\text{CF}_3^+$  makes up  $20 \pm 10\%$  of the total reaction. The channel leading to  $\text{F}_2$  was found to be closed for both ions as might be expected since this necessitates the association of two released fluorine atoms to form the  $\text{F}_2$  molecule.

Previously, a correlation between two H atom loss and exothermicity has been discussed for hydrocarbon ions [17]. For the present systems, it is slightly more exothermic to produce two F atoms for  $\text{CF}_2^+$  than for  $\text{CF}_3^+$ , and more of the three-body channel is found in the former as expected from the exothermicities. The differences are relatively small, both in exothermicity and the amount of events where two F atoms are lost. A comparison to the ratio of three body loss to two body loss for the H atom systems shows no large differences.

The experimental rate coefficient for the DR reaction could be deduced from the signal count rate,  $dN/dt$ , recorded during the scan of the electrons, taking into account the velocity and current of the electrons,  $v_e$  and  $I_e$ , and of the ions,  $v_i$  and  $I_i$  using the expression

$$\langle v_{\text{cm}} \sigma \rangle = \frac{\pi r_e^2}{l} \frac{v_e v_i e^2}{I_e I_i} \frac{dN}{dt}, \quad (3)$$

where  $r_e$  is the radius of the electron beam and  $l$  is the length of the interaction region. The rate coefficient was corrected for contributions to the signal from the regions where the electrons are guided in and out of the straight section of the electron cooler. In these regions the relative collision energy is higher than in the parallel section, due to the increased angle between the velocity vectors of the ions and the electrons [18].

The cross section averaged over the electron velocity distribution was obtained by dividing (3) with the centre-of-mass velocity. The DR cross sections for  $\text{CF}_2^+$  and  $\text{CF}_3^+$  are shown in figure 3. The cross section of  $\text{CF}_2^+$  follows an  $E_{\text{cm}}^{-1.12}$  dependency in the energy range of 1 meV–0.1 eV. The error bars in figure 3 represent the statistical error, but in addition the measurements have a systematic error which arises from the uncertainty in the length of the interaction region, the circumference of the ring, the calibration of the ion current measurement

and the electron density. This amounts to about 15% for the  $\text{CF}_2^+$  measurements performed at CRYRING. The absolute values for the data taken at ASTRID for  $\text{CF}_3^+$  are much less certain, the difference being mainly attributed to the accuracy of the ion current determination. Due to the higher electron velocity spread in the data for  $\text{CF}_3^+$ , the rate coefficient could only be measured for relative energies above 0.01 eV; on the other hand the reaction was studied for reaction energies up to 24 eV.

The two sets of data show a similar behaviour, which is very reminiscent of measurements that were taken using a single pass merged beam technique [19] for ions derived from methane gas ( $\text{CH}_4$ ). In that case, the cross sections for  $\text{CH}_2^+$ ,  $\text{CH}_3^+$  and  $\text{CH}_4^+$  and  $\text{CH}_5^+$  were almost indistinguishable below 0.1 eV, though the  $\text{CH}_3^+$  ions exhibited a steeper energy dependency than  $\text{CH}_2^+$  above this energy. This tendency is also seen in our data here.

It is desirable from a modelling point of view to express the rate coefficients as a function of electron temperature. After unfolding the reaction cross section from the experimental electron temperature distribution, this can be done by convoluting the reaction cross section with a Boltzmann temperature distribution for the electrons:

$$\alpha(T) = \frac{8\pi m_e}{(2\pi m_e k T_e)^{3/2}} \int_0^\infty E_{\text{cm}} \sigma(E_{\text{cm}}) \exp(-E_{\text{cm}}/k T_e) dE_{\text{cm}}, \quad (4)$$

where  $m_e$  is the mass of the electron,  $T_e$  is the electron temperature and  $E_{\text{cm}}$  is the centre-of-mass energy. The rate coefficient for  $\text{CF}_2^+$  was found to be  $3.7 \times 10^{-7} (300/T)^{0.76} \text{ cm}^3 \text{ s}^{-1}$ ,  $20 < T < 1000 \text{ K}$ . Given the uncertainty of the absolute calibration of the  $\text{CF}_3^+$  data, we might assume that the behaviour is similar to that found for  $\text{CH}_2^+$  and  $\text{CH}_3^+$  and thus normalize the cross section for  $\text{CF}_3^+$  to that for  $\text{CF}_2^+$ . This normalization is performed in the energy range of 0.01–0.1 eV. Performing a similar rate coefficient determination for  $\text{CF}_3^+$  yields a value of  $2.6 \times 10^{-7} (300/T)^{0.48} \text{ cm}^3 \text{ s}^{-1}$ ,  $100 < T < 1000 \text{ K}$ . This value is in very good agreement with the value measured in the previous FALP-MS measurement ( $2.8 \times 10^{-7} \text{ cm}^3 \text{ s}^{-1}$  at 300 K [4]), which confirms the procedure. This value is about three times lower than the value that has been used in plasma models [20–23] although the origin of that value is unknown.

In the data obtained for  $\text{CF}_3^+$ , resonant structure is observed starting at around 2 eV and peaking at around 8 eV. A second structure is also seen to begin at about 15 eV though the data do not go to high enough energies to further characterize it. We do not have any information on the highly excited states of  $\text{CF}_3$  and so cannot assign these peaks to specific states. The origin of the resonant structure, however, is thought to be due to electron recombination into Rydberg states converging to excited states of the ion; these states are often repulsive in nature and hence dissociate directly. High energy peaks in recombination cross sections have been seen in a number of theoretical calculations [24–28], and such a feature was seen in our recent measurement of  $\text{CF}^+$  [3]. The structure at around 1 eV in the  $\text{CF}_3^+$  data is not statistically significant, and therefore it is not possible to draw any conclusions concerning that.

## 6. Conclusion

Electron impact ionization of  $\text{CF}_4$  produces the ions  $\text{C}^+$ ,  $\text{F}^+$ ,  $\text{CF}^+$ ,  $\text{CF}_2^+$  and  $\text{CF}_3^+$  with the cross section for the production of the latter being an order of magnitude larger than those for the production of the other individual ions [1]. ( $\text{CF}_4^+$  is unstable and has never been seen.) The recombination rate coefficient for  $\text{CF}^+$  ( $5.2 \times 10^{-8} \text{ cm}^3 \text{ s}^{-1}$  [3]) is lower than those for  $\text{CF}_2^+$  or  $\text{CF}_3^+$ , and reactions of  $\text{CF}^+$  and  $\text{CF}_2^+$  with the  $\text{CF}_4$  parent gas have been shown to produce  $\text{CF}_3^+$  as the only ion product [29]. The dissociative recombination reactions of both  $\text{CF}_2^+$  and  $\text{CF}_3^+$  with electrons are fast processes. The dominant channel (71%) for  $\text{CF}_2^+$  dissociative recombination produces the CF radical and this radical is formed 20% of the time in  $\text{CF}_3^+$

recombination. The  $\text{CF}_2$  radical is formed in the remaining 80% of the  $\text{CF}_3^+$  recombination events. For the production of CF radical, the ionization of  $\text{CF}_4$  followed by recombination competes directly with the electron impact dissociation of  $\text{CF}_4$ , whose cross section is of the same order of magnitude as that for the ionization of  $\text{CF}_4$  [1]. Indeed, experimental studies by Sugai and co-workers [30, 31] indicate that the channel leading to CF from the electron impact dissociation of  $\text{CF}_4$  accounts for only about 15% of the total rate and that leading to  $\text{CF}_2$  only about 10%. Hence, dissociative ionization followed by recombination must be considered the major route for the formation of CF and  $\text{CF}_2$  radicals in etching plasmas. This is in agreement with the conclusions of laser-induced fluorescence studies of the production and loss of this radical in a  $\text{CF}_4$  reactive ion etching plasma [32].

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